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| Division of Forensic Science CONTROLLED SUBSTANCES PROCEDURES MANUAL | Amendment Designator: |
| | Effective Date: 9-December-2003 |
| <p style="text-align: center;">19 NARCOTIC METHODOLOGY</p> <p>19.1 Brief Pharmacology: Analgesic, sedative effects and causes constipation.</p> <p>19.2 Drug Group Examples: Morphine, heroin, hydromorphone, pentazocine, codeine, hydrocodone, oxycodone, methadone, propoxyphene, pethidine (meperidine), and fentanyl</p> <p>19.3 Scheduling:</p> <ul style="list-style-type: none"> • Schedule I – heroin • Schedule II – morphine, oxycodone, hydromorphone, methadone, pethidine (meperidine), fentanyl, codeine (pure), hydrocodone (pure), levomethorphan, 6-monoacetylmorphine (morphine derivative) • Schedule III – Some preparations of hydrocodone or codeine • Schedule IV – pentazocine • Schedule V – Some preparations of codeine (usually cough preparations) • Non-controlled – dextromethorphan • Appropriate caution must be exercised when determining the control status of compounds listed in multiple schedules. Any questions should be answered by consulting appropriate compendia references such as the PDR, Poison Control, DEA Logo Index and the appropriate state or federal codes. Marked capsules or tablets need not be quantitated. Questionable samples require at least a semi-quantitative workup to determine the schedule. <p>19.4 Extraction:</p> <p>19.4.1 Most narcotics may be extracted from basic solutions with organic solvents.</p> <p>19.4.2 Morphine may be extracted from aqueous solution by the addition of a carbonate/bicarbonate buffer and extracting with CHCl_3 or CHCl_3/isopropanol (8:2). Morphine sulfate is not soluble in chloroform.</p> <p>19.4.3 Methadone is often found in orange juice or Tang-type orange drink. The solution should be made basic with sodium carbonate and extracted into CHCl_3 or CH_2Cl_2.</p> <p>19.4.4 Narcotics in cough syrups may be extracted from basic solutions with organic solvents.</p> <p>19.5 Color Test Results: Most narcotics give colors with Marquis, Meckes and Froehdes reagents (see Table 1). Numerous other materials give similar colors, such as methapyriline, glycerol guaiacolate (guaifenesin), and pyrilamine, and are also included.</p> | |

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TABLE 1: Positive Color Test Reactions

| Compound | Marquis | Meckes | Froehdes | HNO ₃ |
|---------------|-----------------|--------------------|---------------------------|------------------|
| Morphine | Purple | Green | Purple | Red |
| Heroin | Purple | Green | Purple | Yellow |
| Codeine | Purple | Blue-green | Green | Yellow |
| Propoxyphene | Black | Orange/brown | Brown | No reaction |
| Meperidine | Orange | Yellow-green | Grey | |
| Pentazocine | Red→olive green | Olive green | Blue | Yellow |
| Hydromorphone | Yellow→red | Yellow-orange | Blue→purple | Yellow-orange |
| Hydrocodone | Purple | Green | Lt. yellow | No reaction |
| Oxycodone | Yellow→purple | Yellow→olive | Yellow | No reaction |
| Methadone | Slow pink | Yellow-green→green | | |
| Methapyrilene | Purple | Purple | Purple | No reaction |
| Guaifenesin | Reddish purple | Green/purple | Green with purple streaks | Yellow |
| Pyrimilamine | Purple | Purple | Purple | |

19.6 TLC:

19.6.1 Baths: TLC1, TLC2, TLC3, TLC4 and TLC5 are recommended.

19.6.2 Detection sprays:

19.6.2.1 Iodoplatinate

19.6.2.2 Ceric Sulfate - Fentanyl may require the use of ceric sulfate as an overspray due to the minute amounts of this material found in most pharmaceutical preparations.

19.7 UV:

19.7.1 Not usually unique to the specific compound.

19.7.2 Results:

- Morphine – max. at 285 nm in acid
- Heroin – max. at 280 nm in acid (Heroin will often hydrolyze to monoacetylmorphine and morphine with prolonged exposure to aqueous solutions.)
- Methadone – max. at 250 and 292 nm in acid
- Pethidine (Meperidine) – triplet - max. at 258 nm in acid

19.8 Dextromethorphan Enantiomer Determination

19.8.1 Because of the scheduling differences between levomethorphan (Schedule II) and dextromethorphan (non-controlled), a microcrystal test must be performed for differentiation unless found in a recognizable pharmaceutical preparation.

19.8.2 Materials:

- Glass slides and coverslips
- Polarizing microscope
- 10% Platinic Chloride solution in water (w/v)
- 1% acetic acid solution in water

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| <div data-bbox="342 266 782 296"> <ul style="list-style-type: none"> Dextromethorphan reference standard </div> <div data-bbox="240 327 448 357"> <p>19.8.3 Procedure:</p> </div> <div data-bbox="334 388 1544 1087"> <p>19.8.3.1 Place one drop of 1% acetic acid in water to a small portion (less than 1mg of pure dextromethorphan is needed) of the dextromethorphan standard on a glass slide. Then add a portion of a drop of 10% platinum chloride in water and cover. Feathery dendrites will form within 2 minutes.</p> <p>19.8.3.2 Place one drop of 1% acetic acid in water to a small portion of the sample on a glass slide. Then add a portion of a drop of 10% platinum chloride in water and cover. Feathery dendrites will form when the sample is a pure enantiomer. A racemic mixture will not form these microcrystals.</p> <p>19.8.3.3 To verify the identity as the dextro isomer, mix a small amount of sample with approximately the same amount of dextromethorphan reference standard on a glass slide. Add the acetic acid and platinum chloride solutions as stated above. Observe any microcrystalline formation. If the sample is dextromethorphan, the feathery dendrites will form; if the sample is levomethorphan or racemethorphan, no dendrite-shaped crystals will form.</p> <p>19.8.3.4 The microcrystal formations should be contemporaneously verified or photographed. Verification must be documented in the case notes with the initials of the verifier and the date.</p> <p>19.8.3.5 The sample may need to be purified to allow for crystal formation. Mixtures of dextromethorphan and MDMA will need to be separated before the crystal test, as pure MDMA reacts with platinum chloride to form similar, but not the same, microcrystals.</p> <p>19.8.3.6 Reference: Fulton, Charles C. <i>Modern Microcrystal Tests for Drugs</i> , New York: Wiley-Interscience, 1969, pp. 58-59.</p> </div> <div data-bbox="152 1119 483 1148"> <p>19.9 Codeine Quantitation</p> </div> <div data-bbox="240 1180 904 1209"> <p>19.9.1 See GC section 10 for general quantitation procedure.</p> </div> <div data-bbox="240 1241 438 1270"> <p>19.9.2 Reagents:</p> </div> <div data-bbox="342 1302 753 1396"> <ul style="list-style-type: none"> Methylene Chloride or Chloroform Dicyclohexylphthalate (DCHP) Codeine Standard </div> <div data-bbox="240 1428 620 1457"> <p>19.9.3 Internal Standard Solution:</p> </div> <div data-bbox="334 1488 1451 1610"> <p>19.9.3.1 Prepare a sufficient volume to dilute the codeine standard solution and all samples.</p> <p>19.9.3.2 Prepare a 1.5 mg/mL solution of DCHP in methylene chloride or chloroform in an appropriate volumetric flask.</p> </div> <div data-bbox="240 1642 636 1671"> <p>19.9.4 Codeine Standard Solutions:</p> </div> <div data-bbox="334 1703 1503 1885"> <p>19.9.4.1 Weigh 20 mg of Codeine and transfer to a 10 mL volumetric flask with internal standard solution. Dilute to mark with internal standard solution. This results in a solution for 2.0 mg/mL codeine in internal standard solution.</p> <p>19.9.4.2 Prepare a solution of another concentration within the linear range in the same manner to use as the check standard.</p> </div> <div data-bbox="240 1917 547 1946"> <p>19.9.5 Sample preparation:</p> </div> | |

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| <div data-bbox="334 291 1494 354">19.9.5.1 Verify the identity of the salt originally present in the sample by suitable chemical testing. Adjust sample weight accordingly.</div> <div data-bbox="334 386 1511 449">19.9.5.2 Weigh approximately 20 mg of the sample and made basic with 4N sodium hydroxide solution until pH is above 10.</div> <div data-bbox="334 480 1373 512">19.9.5.3 Extract with a known volume of internal standard solution in a small separatory funnel.</div> <div data-bbox="334 543 1503 575">19.9.5.4 Extraction is necessary in order to remove the salt for comparison to the Codeine standard solution.</div> <div data-bbox="240 606 500 638">19.9.6 GC parameters:</div> <div data-bbox="342 669 1211 764"> <ul style="list-style-type: none"> • Column: 15 m HP-1 or HP-5 capillary (0.25 mm i.d., 0.25 µm film thickness) • Oven temperature: 220-240° C • FID temperature: 280° C </div> <div data-bbox="240 795 659 827">19.9.7 Codeine elutes prior to DCHP.</div> <div data-bbox="151 858 472 890">19.10 Heroin Quantitation</div> <div data-bbox="240 921 902 953">19.10.1 See GC section 10 for general quantitation procedure.</div> <div data-bbox="240 984 436 1016">19.10.2 Reagents:</div> <div data-bbox="342 1047 753 1142"> <ul style="list-style-type: none"> • Methylene Chloride or Chloroform • Dicyclohexylphthalate (DCHP). • Heroin Standard </div> <div data-bbox="240 1173 618 1205">19.10.3 Internal Standard Solution:</div> <div data-bbox="334 1236 1313 1268">19.10.3.1 Prepare a sufficient volume to dilute the heroin standard solution and all samples.</div> <div data-bbox="334 1299 1451 1352">19.10.3.2 Prepare a 1.0 mg/mL solution of DCHP in methylene chloride or chloroform in an appropriate volumetric flask.</div> <div data-bbox="240 1383 618 1415">19.10.4 Heroin Standard Solutions:</div> <div data-bbox="334 1446 1500 1541">19.10.4.1 Weigh ~20 mg of Heroin and transfer to a 10 mL volumetric flask with internal standard solution. Dilute to mark with internal standard solution. This results in a solution of ~2.0 mg/mL Heroin in internal standard solution.</div> <div data-bbox="334 1572 1503 1625">19.10.4.2 Prepare a solution of another concentration within the linear range in the same manner to use as the check standard.</div> <div data-bbox="240 1656 550 1688">19.10.5 Sample Preparation:</div> <div data-bbox="334 1719 907 1751">19.10.5.1 Weigh 10-40 mg of sample into a test tube.</div> <div data-bbox="334 1782 1094 1814">19.10.5.2 Dilute with internal standard solution to appropriate volume.</div> <div data-bbox="240 1845 500 1877">19.10.6 GC parameters:</div> <div data-bbox="342 1908 1211 1961"> <ul style="list-style-type: none"> • Column: 15 m HP-1 or HP-5 capillary (0.25 mm i.d., 0.25 µm film thickness) • Oven temperature: 260-280° C </div> | |

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| <div> <div> <ul style="list-style-type: none"> • FID temperature: 280° C </div> <div> <p>19.10.7 DCHP elutes prior to Heroin. Peaks between DCHP and Heroin are often due to monoacetylmorphine or acetylcodeine.</p> </div> <div> <p align="right">◆ End</p> </div> </div> | |